



Suppression of hydrogen-induced damage in friction stir welded low carbon steel joints



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ABSTRACT

Hydrogen-induced damage including blister and cracking in friction stir welded low carbon steel were evaluated by the cathodic hydrogen charging method. After hydrogen charging for 2 h, irreversible dome-shaped blisters and internal cracking began to appear on the surface of the base metal. However, after hydrogen charging for 16 h, cracking formed along the thermo-mechanically affected zone boundary, while the blisters or cracking were hardly observed in the stir zone. In addition, the stir zone showed a plasticity reduction from 38% to 28%, much less than that of the base metal reduced from 48% to 2%.

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1. Introduction

Hydrogen embrittlement (HE) is defined as the deterioration of the mechanical properties resulting from the introduction of hydrogen into the materials by various methods including hydrogen-induced cracking (HIC), hydrogen blistering, stress corrosion cracking (SCC), etc. HE is a long-term problem, which has widely occurred during solidification, heat treatment, or a cathodic reaction. A special case is fusion welding, in which the atomic hydrogen formed during welding dissolves in the liquid metal of the weld. After solidification, some of the dissolved hydrogen will precipitate and often leads to the formation of cracks in the heat affected zone (HAZ) adjacent to the weld. It was found that absorbed atomic hydrogen migrating in the steel is accumulated on the fronts of non-metallic inclusions, microcracks or grain boundaries, and the recombination of atomic hydrogen into the molecular form causes an increase in the pressure of nascent H₂ bubbles as well as nucleation and the development of microcracks. Opiela et al. studied the HE behavior of welded joints by cathodic hydrogen charge, and found that the welded joints are susceptible to hydrogen cracking in both the HAZ and fusion zone [1–3]. It was postulated that when hydrogen has entered a steel structure, the mechanism causing hydrogen embrittlement is the same, regardless of the hydrogen source. The microstructure of the steel is the main factor that dominates the susceptibility of the material to HE. Although the structural characteristics of the materials that significantly influence the hydrogen induced cracking process are still not clearly understood,

it is known that the phase constitution, grain boundary character and crystallographic texture, etc., affect the generation and propagation of hydrogen-induced crack in steel materials [4–7]. Generally, two of the most established mechanisms of hydrogen-induced failure of the materials are the hydrogen-enhanced local plasticity model (HELP) and the hydrogen-enhanced decohesion model (HEDE). The kind of mechanism that occurs during fracture strongly depends on the microstructure of the materials. For example, the fully ferritic steels essentially fracture through the HELP effect [8], while lath martensite steel was reported to undergo HELP associated with the HEDE effect [9]. Given the more complex microstructure, the HE behavior of the material maybe correspondingly complicated.

Friction Stir Welding (FSW) was invented by the Welding Institute (TWI) of the UK in 1991 with the original purpose of joining Al and Al alloys, since Al alloys are very difficult to weld by conventional fusion welding methods [10]. FSW is a solid-state joining technique, in which a rotating tool is plunged into the work-pieces and traverse along the weld path. In this way, the rotating tools can plastically deform (stir) and transport the surrounding material from the front to the back of the tools. As a result, the work-pieces can be stirred together to form a joint [11,12]. With the development of the FSW technique and the highly durable rotating tools, this technique has been expanded to many other high melting points metallic materials including Cu, Ti, Fe and steels [13–15]. Recently, the FSW technique has been successfully used for the welding of high carbon steels, which are considered as unweldable materials by fusion welding methods due to the formation of the brittle martensitic phase [16,17]. Because of the complex materials flow during the FSW process [18], the resultant microstructure in

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the welded area is very complicated and significantly depended on the welding conditions and the material properties. For example, a very refined microstructure can be obtained in the stir zone (SZ) due to recrystallization. Since the FSW is carried out in the solid state, the problems caused by the dissolution of hydrogen into the materials may be significantly attenuated. Therefore, the friction stir welded joint must show different HE behavior comparing with its fusion welded counterpart. Recently, Khodir et al. first studied the blister suppression in a kind of high carbon steel by friction stir processing [19]. The prevention of the blister formation was proposed to be caused by the microstructure refinement and the prevention of formation of hard carbide particles in the soft ferrite matrix. After that, the HE behavior of the FSW processed steels has hardly been studied.

Though it is generally regarded that the hydrogen-induced embrittlement is a problem associated with high strength steel, in fact, a great number of studies has revealed that from low carbon steels to even high purity iron, they are all susceptible to the detrimental effect of hydrogen [20–26]. In this study, the sensitivity of the FSW processed low carbon steel to HE is investigated by the hydrogen cathodic charging method. The hydrogen induced damage including surface blistering and hydrogen-induced cracking is thoroughly investigated. The mechanical property of the SZ related to the blister or surface crack formation is investigated and compared with that of the base metal (BM) and the mechanism for hydrogen induced mechanical degradation is illustrated and discussed.

2. Experimental details

2.1. Materials

In this study, the as-received low carbon steel plates with a thickness of 0.8 mm were subjected to the FSW process using a load-controlled FSW machine. The chemical composition of the steel plate is shown in Table 1. A pin-less rotating tool made of Si_3N_4 ceramic, which had a shoulder diameter of 12 mm, was used during the welding process. The tool axis was tilted by 2° with respect to the normal direction of the sample surface. The traveling speed of the rotating tool was kept constant at 280 mm/min and the rotation speeds was 320 rpm. Argon shielding gas was used during the welding process to protect the SZ from oxidation.

2.2. Cathodic hydrogen charging

Fig. 1(a) shows the typical appearance of the friction stir welded sample. After welding, the dog-bone like tensile specimen and rectangular specimens for microstructural characterization were cut by an electrical discharge machine from the FSWed plates parallel to the welding direction, which are illustrated in Fig. 1(b). The rectangular specimen consisted of all the specific zones including the BM, HAZ, thermo-mechanically affected zone (TMAZ) and SZ, while the tensile specimen had a gauge length of 6 mm, width of 3 mm and thickness of about 0.8 mm. Thus the tensile specimen was completely inside the area of the SZ. Prior to hydrogen charging, the entire surface of the specimens was mechanically ground using emery papers to 4000 grade followed by polishing with a col-

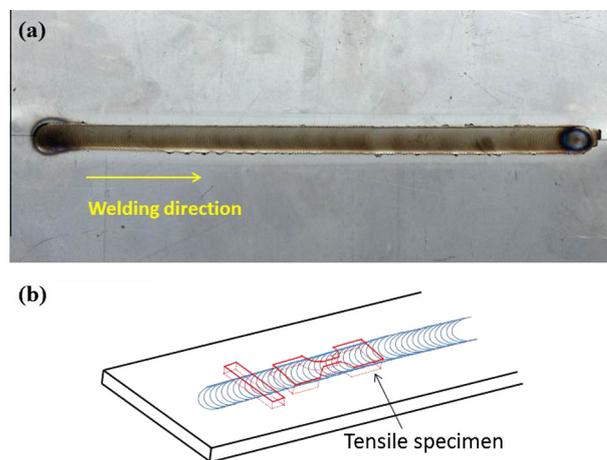


Fig. 1. (a) Appearance of the friction stir welded plates; and (b) schematic map showing the specimen preparation method.

loidal silica oxide polishing suspension, then rinsed and degreased with acetone. A thin stainless wire was electric resistance spot welded to the specimen which was placed into a charging cell filled with an aqueous solution of 0.5 M H_2SO_4 containing 1 g/L thiourea and surrounded by a Pt wire. Since the specimen and the Pt wire were immersed into the solution in the cell, Pt dissolution and subsequent Pt re-deposition on the specimen might occur during the hydrogen charging. However, the Pt deposition exerted similar effect on the hydrogen charging behavior of the BM and SZ. The effect was very small and therefore was not considered in the present study. Hydrogen was introduced into the specimens by cathodic charging at the current density of 500 A/m^2 using a regulated direct current power supply under ambient temperature. For comparison, the specimens were hydrogen charged for different periods of 2, 4, 8 and 16 h. After charging, the surfaces of the charged specimen were still very shiny and hydrogen blisters could be visually seen. The specimens were then immediately washed with distilled water and acetone.

In order to determine the hydrogen content charged into the specimen, a Horiba EMGA-830 hydrogen content analyzer was employed for melt extraction. At least three specimens weighing about 0.2 g each were tested immediately after the hydrogen charging. The specimens were quickly heated up to their melting points temperatures for evaporating all gases, which were then taken by a nitrogen carrier gas through chemical reaction tubes to the analyzing units. The thermal conductivity of the carrier gas was measured and the software then calculated the hydrogen concentration of the sample based on the thermal conductivity variation. The estimated value represents the total amount of hydrogen in the material. However, very small amount of hydrogen may be lost in the process of sample preparation before being subjected to the hydrogen measurement.

2.3. Microstructure and mechanical properties evaluation

The microstructures of the FSWed joints as well as the BM before and after hydrogen charging were observed by optical microscopes (OM), a scanning electron microscope (SEM) attached with an electron backscatter diffraction (EBSD) system and a transmission electron microscope (TEM). For OM and SEM observation, the samples were mechanically polished followed by chemical etching using 3% Nital solution. For the TEM observations, the samples were prepared by twin-jet electropolishing with a solution of $\text{HClO}_4:\text{CH}_3\text{COOH} = 1:9$. The tensile testes for the uncharged and charged specimens were carried out using an Instron tensile

Table 1
Chemical composition of low carbon plates (wt.%).

Steel type	Chemical composition (mass%)				
	C	Mn	P	S	Fe
Low carbon steel	0.12	0.50	0.40	0.045	Bal.

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